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METHOD AND DEVICE FOR DETERMINING THE THICKNESS AND/OR COMPLEX REFRACTIVE INDEX OF THIN LAYERS AND THE USE THEREOF FOR CONTROLLING COATING PROCESSES

Harald Schulz et al.

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METHOD AND DEVICE FOR DETERMINING THE THICKNESS AND/OR COMPLEX REFRACTIVE INDEX OF THIN LAYERS AND THE USE THEREOF FOR CONTROLLING COATING PROCESSES

Inventor; and Inventor/Applicant (for US only):	Harald Schulz et al.
Applicant (for all contracting states except US):	Wissenschaftlich-Technisches Optikzentrum Nordrhein-Westfalen (Optikzentrum NRW) E.V.

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The invention pertains to a method and device for determining the thickness and/or the complex refractive index of one or more thin layers that are applied to substrates in order to endow the substrates with new properties, as well as the use of said method and device for controlling coating processes. /1*

It is known to utilize crystals for measuring layer thicknesses during coating processes. In this case, the natural frequency of the crystal is detuned due to the changes in mass caused by the coating.

It is also known to utilize occurring optical interference for determining the thickness of such layers. In this case, the change in the reflected luminous intensity is evaluated over the coating duration at a single wavelength λ. The reflected luminous intensity changes due to the increase in the optical path $P = 2 * n_R * d$ caused by the increasing layer thickness. At a certain layer thickness, the light beam reflected by the substrate interferes with the part of the light beam that is reflected on the layer surface. The destructive interference which corresponds to the minimum reflected luminous intensity can be determined by $P = \lambda/2$. If the refractive index n_R is known, the layer thicknesses d, in the presence of a destructive interference, is defined as $d = \lambda/(4 * n_R)$. /2

When determining the thicknesses of several layers that may also be arranged on top of one another, it is necessary to use a new uncoated glass sample for each individual layer of the multi-layer system.

Although known methods and devices of this type make it possible to measure individual layers, it is impossible to sequentially measure the entire applied layer system.

In addition, another measuring device is required for determining the complex refractive index $n = n_R + i * n_i$ that influences the optical properties. In this case, ellipsometers are normally utilized outside the coating system for determining the reflection and the transmission by measuring the angular resolution.

The invention is based on the objective of developing a method and device which ensure a corresponding simplicity of operation, allow the measurement of multiple layer systems in this form and make it possible to carry out measurements in situ, wherein the obtained measurements can also be used in controlling the coating process. /3

According to the invention, this objective is attained with the method disclosed in the characterizing portion of Claim 1 and the device disclosed in the characterizing portion of Claim 8. Advantageous embodiments and additional refinements of the invention are defined by the characteristics contained in the subordinate claims.

* [Numbers in the margin indicate pagination of German text.]

In the method according to the invention, the practically simultaneous wavelength-resolved and/or polarization-resolved measurements of the light reflected and the light transmitted by the coated substrate make it possible to determine the thickness and/or the complex refractive index of layers in multi-layer systems ex-situ as well as in-situ during the coating of the substrate.

In this case, the entire spectral range of the reflection R (λ) and the transmission T (λ) is determined virtually simultaneously. Although not absolutely imperative, this may also be carried out in a polarization-resolved fashion by polarizing twice, namely perpendicular and parallel, and by measuring the portions R_s (λ), R_p (λ), T_p (λ), T_s (λ) which are polarized perpendicular and parallel in a virtually simultaneous manner.

The individual layers of multi-layer systems can be characterized by comparing the measured reflection R_{im} (λ) and transmission T_{im} (λ) with respective reflection and transmission values that are theoretically calculated for the respective wavelengths. In this case, the index i refers to the respective reflection and transmission values after applying the ith layer. The comparison between all theoretically calculated values and the measured spectral values is carried out for all evaluated wavelengths λ_j, where j = 1, 2, 3 ... m. The number of evaluated wavelengths m should be greater than 30, preferably in the range above 250 different wavelengths λ_j. A number of light-sensitive detectors which corresponds to the number m of different wavelengths to be evaluated must be arranged in a detector one-dimensionally or two-dimensionally such that they are able to measure the individual spectral lines generated by means of a dispersive element. This number can be chosen in accordance with the required measuring accuracy or the available detector. /4

In addition to a comparison to theoretically calculated values, it is also possible to carry out an evaluation in the form of a nominal value/actual value comparison in which the measured values are compared with calibration values that are determined beforehand on known samples. The theoretically calculated values, as well as the calibration values, may be stored in a database or in an evaluation unit and made available for the comparison with the measured values.

The theoretical values R_i (λ_j) and T_i (λ_j) can be determined by defining the refractive index and the nominal layer thickness. The three parameters d (layer thickness), n_R and n_i (complex refractive index) can be determined by evaluating a relatively large number of wavelengths λ_j over a broad spectral range (UV- NIR). This can be realized by converting a matrix equation system as described in greater detail below. It is preferred to select wavelengths for this determination which have a low dependence on the refractive index. In contrast to the conventional utilization of interferometers, the method according to the invention makes it possible to measure the reflection and the transmission characteristics of a glass sample that is successively coated with several layers, and subsequently to compare these characteristics with /5

the theoretically calculated values or the calibration values. In this way, a correspondingly coated glass sample need not be exchanged during the measurements, and the characteristic of the entire layer system can be determined.

If the method according to the invention is used in controlling a coating system, the deviations of the layer thickness and the refractive index from predetermined nominal values can be minimized over the entire spectral range of interest.

The characteristics R_i and T_i can be calculated with a matrix algorithm, for example, as described in M. Born, E. Wolf: "Principles of Optics" Pergamonpress; Oxford, 1980. In this case, each individual layer of a multi-layer system is described in the form of a complex 2 x 2 matrix M_i. The parameters of the matrix are the layer thickness d, the real portion of the refractive index /6
n_R and the imaginary portion of the refractive index n_i.

For example, if 256 different wavelengths are used for determining the layer characteristic, a comparison between each individual measured wavelength and the stored calculated value or previously determined calibration value is carried out in a wavelength-resolved manner:

$$\sum_{j=1}^{256} [T_i(\lambda_j) - T_{im}(\lambda_j)]^2/256 = Min$$

$$\sum_{j=1}^{256} [R_i(\lambda_j) - R_{im}(\lambda_j)]^2/256 = Min$$

and in a polarization-resolved manner:

$$\sum_{j=1}^{256} [T_{ip}(\lambda_j) - T_{ipm}(\lambda_j)]^2/256 = Min$$

$$\sum_{j=1}^{256} [T_{is}(\lambda_j) - T_{ism}(\lambda_j)]^2/256 = Min$$

$$\sum_{j=1}^{256} [R_{ip}(\lambda_j) - R_{ipm}(\lambda_j)]^2/256 = Min$$

In this case, it is advantageous that corrective measures can be initiated during the coating process if it is determined that a layer /7

$$\sum_{j=1}^{256} [R_{is}(\lambda_j) - R_{ism}(\lambda_j)]^2/256 = Min$$

in the layer system being applied does not fulfill the specified requirements. Such a defect can be corrected by changing the corresponding parameter in at least one of the subsequently applied layers.

The mathematical algorithm used for calculating the reflection and transmission characteristics is described in greater detail below. In this case, the substrate material, the entry medium and the exit medium, as well as the number of layers, their sequence and the corresponding nominal layer thicknesses, are input first. The matrix is subsequently calculated for each individual layer with the aid of corresponding values that are specific for each material and stored in a material file or, as described above, with measured and stored calibration values $m_R(\lambda_j)$, $n_i(\lambda_j)$. The reference numeral M_i identifies the matrix of the ith layer, and the matrix of a layer system M_i^* can be determined for i layers by

$$M_i^* = M_i \;\ldots\ldots\; M_3 * M_2 * M_1 = \begin{matrix} a_i & b_i \\ c_i & d_i \end{matrix}$$

The method according to the invention can be carried out with a measuring device in which an essentially collimated light beam of a light source that is preferably realized in the form of a white light source is used. This makes it possible to also use the measuring device directly in the coating system. The emitted light beam is preferably guided through a protective tube with a high length to diameter ratio in order to protect the entire lens system from the coating material. /8
The relatively small diameter of the protective tube causes a slight angular tolerance for the angle of incidence of the light reflected on a glass sample.

A highly collimated white light source with a high luminous intensity in the form of a halogen reflector lamp is preferably utilized for a device according to the invention, wherein the halogen reflector lamp has a parabolic reflector and an axial filament which maintain the aperture angle of the light cone in the range between 5° and 12°. The spectral characteristic of such a light source $L(\lambda_j)$ can be influenced together with the spectral characteristic of a detector $D(\lambda_j)$ by additionally coating the halogen reflector lamp, such that the lowest possible spectral variation occurs:

$L(\lambda_j) * D(\lambda_j) \approx L(\lambda k) * D(\lambda k)$ for $j \neq k$; this makes the dynamic of the measurement independent of the wavelength. The light source used is preferably operated with a DC voltage such that a high constancy of the light emission over a certain duration is achieved.

The light beam emitted by the light source is collimated and subsequently focused into a fiber optic waveguide.

In this case, a drawn fiber optic waveguide that is conically tapered in the beam direction is preferably utilized. This serves for utilizing the total reflection on the fiber casing in such a way that the light emerging from the conical fiber optic waveguide is focused on a diameter of approximately 100 µm-300 µm. The emitted light beam is divided by means of a Y-coupler, /9
wherein one part of the light beam is directed onto the sample and another part is directed onto a detector. Instead of utilizing a Y-coupler, the light beam may, for example, also be divided with two fiber optic waveguides that are arranged closely adjacent to one another.

The conically tapered fiber optic waveguide provides the advantage that the transmitted light has a high luminous density.

The fiber optic waveguide that is inserted into a transmitting unit is fixed thereon by means of a conventional plug-type fiber connector, and the emerging light is re-collimated in order to obtain a parallel light beam. Converging or diverging light can be adjusted by varying the spacing between the transmitting unit and the sample or the curvature radius of the sample by replacing different lenses in the transmitting unit.

The light that is incident on the sample is partially reflected and transmitted into an additional fiber optic waveguide in a receiving unit by means of another beam splitter and additional collimating lenses, wherein the additional fiber optic waveguide transmits the reflected light R to a multiplexer. The light transmitted through the sample is also transmitted to the multiplexer through a fiber optic waveguide by means of collimating lenses of a second receiving unit, wherein the reflected light R, the transmitted light T and the emitted light L are combined in said multiplexer. In this case, the multiplexer is designed in such a way that it respectively directs one part of this light L (λ_j), R (λ_j) and T (λ_j) onto a fiber-coupled spectrometer intermittently in a /10
virtually simultaneous fashion.

In the wavelength-resolved measurement, the multiplexer has a triple input with three fiber optic waveguides that preferably have a diameter of approximately 500 µm and a single output fiber leading to the spectrometer that is realized in the form of a dispersive element. In order to prevent coupling losses, the three fiber optic waveguides of the multiplexer are arranged at a distance of less than 1 mm from the fiber optic waveguides L, R and T. For example, electromechanical or electroöptical shutters may be arranged in this relatively small intermediate space. These shutters make it possible to respectively open any one of the three channels and to block the other channels so that only the light emitted from one fiber optic waveguide is always directed onto the detector.

In this case, it is particularly advantageous to realize the electromechanical shutters or other equivalently acting means in such a way that the time at which each channel is opened and the opening duration can be separately adjusted. This makes it possible to adapt this time to the integration time of the detector in the spectrometer, which preferably can also be adjusted individually for each channel. In this way, it is possible to achieve an optimal amplitude of the detector depending on the type of coating (antireflective, highly reflective) and the corresponding intensity of the reflected/transmitted luminous energy. The integration time preferably can be variably adjusted within a range of approximately 100-500 ms such that a virtually simultaneous measurement of the three channels is possible.

In another embodiment of the method according to the invention, the reflected and the /11
transmitted light are measured in a polarization-resolved manner. In this case, polarizers and analyzers are used for generating and subsequently evaluating the part of the light that, for example, is polarized perpendicular and parallel. If the light is polarized parallel and

perpendicular, a multiplexer with five different channels must be utilized instead of a multiplexer with three channels. The polarization-resolved measurement is particularly suitable for characterizing thin-film polarizing layers.

The invention is described in greater detail below with reference to embodiment examples.

The figures show:

Figure 1 is a block diagram of a device for carrying out a wavelength-resolved measurement;

Figure 2 is a device for carrying out a polarization-resolved measurement, and

Figure 3 is a special construction for the device according to the invention.

A light source 1 that is realized in the form of a white light lamp and contains a parabolic reflector 2 emits light into a conically tapered fiber optic waveguide 4 via a collimating lens 3. The light is transmitted to a Y-coupler 7 via another fiber optic waveguide 6 and divided in said coupler, wherein one part of the light is directly transmitted to a triple multiplexer 15 by means of /12
a fiber optic waveguide L, and wherein the other part of the light beam is transmitted to a transmitting unit in which the corresponding fiber optic waveguide is held by means of a plug-type fiber connector 8. The transmitting unit directs the corresponding part of the light beam onto a sample 12 by means of a collimating lens 9, a beam splitter 10 and a protective shield 11. The protective shield 11 is preferably inclined at an angle that is greater than 0° and less than approximately 10° in order to prevent reflections toward the fiber optic input waveguide and to keep the light transmission independent of the polarization.

The light is surrounded by a protective tube 5 with a high aspect ratio between the protective shield 11 and the sample 12.

The part of the light which is reflected by the sample 12 is deflected with the aid of the beam splitter 10. Another collimating lens 17 arranged in the transmitting unit transmits this part of the light into another fiber optic waveguide R that, in turn, transmits the light to the multiplexer 15.

Another collimating lens 13 is arranged underneath the sample 12. This collimating lens transmits the light transmitted through the sample 12 to the multiplexer 15 via the fiber optic waveguide T that is also attached by means of a plug-type fiber connector 14.

The figure schematically indicates that the multiplexer 15 contains three inputs L, T and R, and that coupling points for each of these inputs are provided on the multiplexer. The multiplexer 15 contains an output for transmitting the light to a detector 16 that contains a spectrometer (preferably a grating spectrometer) and a corresponding quantity of light-sensitive detectors. In this case, the detectors must be arranged such that a wavelength-resolved measurement is possible. Electromechanical shutters that are not illustrated in the figure may be /13

arranged in the multiplexer 15. These shutters are able to open and close the outlet openings of the fiber optic waveguides L, R, and T, namely in such a way that only one of the channels is respectively opened. The measuring values of the light-sensitive detectors are fed to an evaluation and control unit 18, in which a nominal value/actual value comparison is carried out with theoretically calculated values or calibration values that are stored in a memory arranged in the evaluation and control unit. The arrow on the evaluation and control unit 18 is intended to indicate the option of directly utilizing the result of the comparison in controlling the coating process. The generated control signal may be used particularly in reactive processes. For example, if an insufficient reactive partial pressure is present, timely detection makes it possible to determine whether or not the layer has the required stoichiometry. When applying silicon oxide layers, it is, for example, possible to determine whether silicon monoxide or silicon dioxide was formed, namely because silicon monoxide has a higher absorption than silicon dioxide. This is undesirable in many applications.

Figure 2 shows an example for a polarization-resolved measurement. In this case, identical elements are identified by the same reference numerals as in Figure 1.

One part of the light transmitted by the light source 1 is directly transmitted to a five-fold multiplexer 19, which, except for the number of input channels, corresponds to the multiplexer 15 /14 used in the embodiment according to Figure 1 via a fiber optic waveguide L. Another part of the light emitted by the light source 1 is directed onto the sample 12 via a collimating lens 3 and a polarizer 20, where the light is polarized parallel and perpendicular. The polarized light is incident on the sample 12 at a certain angle such that a beam splitter is not required as was the case in the embodiment according to Figure 1. An analyzer 21 is arranged at an angle of reflection that corresponds to the angle of incidence of the polarized light in order to receive the light reflected by the sample 12. The analyzer 21 separates the polarized light into the part that is polarized parallel and the part that is polarized perpendicular and transmits these parts into fiber optic waveguides R_p and R_s by utilizing lenses that are illustrated merely schematically in this figure. These two fiber optic waveguides R_p and R_s transmit the light to the multiplexer 19.

In order to divide the part of the light that is transmitted through the sample 12, another analyzer 21 is arranged in accordance with the orientation of the light transmitted by the light source 1 and divides the light components that are polarized perpendicular and parallel. These two light components are also transmitted to the multiplexer 19 by means of two additional fiber optic waveguides T_p and T_s.

The multiplexer operates analogouslyly to the previously described multiplexer 15. The only difference between the two multiplexers is that five channels must be opened and closed instead of only three channels. The part of the light is intermittently transmitted to the detector 16

that is also realized analogously to the detector described with reference to Figure 1. The individual measuring values are sent for additional processing by the detector 16 as schematically /15 indicated by the arrow. In this case, a nominal value/actual value comparison is also carried out, and the results can also be used for controlling the coating process.

Figure 3 shows the structure of a measuring device, in which the angular tolerance for the angle of incidence of the light reflected on the sample 12, in connection with the relatively small diameter of the protective tube 5, is observed in a particularly favorable fashion by utilizing six rods (hexapods).

Claims /16

1. Method for measuring the thickness and/or the complex refractive index of thin layers applied to substrates, where the reflection R and the transmission T of a light beam L that is directed onto a sample (12) to be evaluated are measured virtually simultaneously in a wavelength-resolved and/or a polarization-resolved fashion.

2. Method according to Claim 1, characterized by the fact that the measured values of the reflection R_{im} (λ_j) and the transmission T_{im} (λ_j) for a layer i to be evaluated are compared with calculated values for the theoretical reflection R_i (λ_j) and the theoretical transmission T_i (λ_j) for different wavelengths λ_j of the transmitted light L which are stored in a database or corresponding calibration values that are determined in comparison measurements, namely in the form of a nominal value/actual value comparison.

3. Method according to Claim 1 or 2, characterized by the fact that the measured reflection R_{im} and the measured transmission T_{im} are measured and compared for a large number of wavelengths λ_j over a broad spectral range.

4. Method according to at least one of Claims 1-3, characterized by the fact that the wavelengths λ_j are selected with little dependence on the refractive index.

5. Method according to least one of Claims 1-4, characterized by the fact that the /17 spectrum of the light L (λ_j) that is directed onto the sample (12) is also measured.

6. Method according to at least one of Claims 1-5, characterized by the fact that the spectra of the incident light L (λ_j), the reflected light R (λ_j) and the transmitted light T (λ_j) are measured intermittently but simultaneously with a spectrometer.

7. Method according to Claim 6, characterized by the fact that the measuring time of the individual components of the incident light L, the reflected light R and the transmitted light T are varied such that optimal integration conditions are achieved.

8. Device for carrying out the method according to Claim 1, characterized by the fact that a light source (1) is provided, wherein the light from said light source which has a broad wavelength range is transmitted to a detector (16) via fiber optic waveguides (4, 6, L, R, T,

$R_{illegible}$, R_s, T_p, T_s) after passing through a multiplexer (15, 19) that intermittently opens its respective channels, wherein one part of the light is directly transmitted to the multiplexer (15, 19), and wherein the part of the light that is reflected by a sample (12) and the part of the light that is transmitted through the sample (12) are separately transmitted to the detector (16) by the multiplexer (15, 19).

9. Device according to Claim 8, characterized by the fact that the light source (1) consists /18 of a white light source.

10. Device according to Claim 8 or 9, characterized by the fact that the light of the light source (1) can be transmitted into a conically tapered fiber optic waveguide (4).

11. Device according to Claim 10, characterized by the fact that the fiber optic waveguide (4) is surrounded by a protective tube (5) with a high length-to-diameter ratio.

12. Device according to Claim 11, characterized by the fact that the protective tube (5) is metal.

13. Device according to Claim 11, characterized by the fact that the fiber optic waveguide (6) is surrounded by a polyimide jacket.

14. Device according to one of Claims 8-13, characterized by the fact that the light beam can be divided by means of a Y-coupler (7) before it is incident on the sample (12).

15. Device according to at least one of Claims 8-14, characterized by the fact that the lens systems are protected with protective shields.

16. Device according to Claim 15, characterized by the fact that the protective shields are inclined relative to the light beam by an angle that is greater than 0° and smaller than approximately 10°.

17. Device according to at least one of Claims 8-16, characterized by the fact that the /19 detector (16) is formed by a dispersive element and a one-dimensional or two-dimensional field of light-sensitive detectors that are arranged in a defined fashion.

18. Device according to Claim 17, characterized by the fact that the dispersive element is a spectrometer.

19. Device according to Claim 18, characterized by the fact that the dispersive element is a grating spectrometer.

20. Device according to Claim 9, characterized by the fact that the light source (1) contains a parabolic reflector (2) that ensures an aperture angle of the light cone between 5-10°.

21. Device according to at least one of Claims 8-20, characterized by the fact that the multiplexer (15, 19) contains electromechanical or electroöptical shutters that make it possible for the multiplexer separately to receive the light L emitted by the light source (1), the light R reflected by the sample (12) and the light T transmitted through the sample (12).

22. Device according to at least one of Claims 8-20, characterized by the fact that one part of the [light emitted] by the light source (1) can be directed onto the sample (12) by means of a polarizer (20), and by the fact that the light reflected by the sample (12) and the light transmitted through the sample can be transmitted to the multiplexer (19) while being divided into at least /20
two differently polarized components by means of analyzers (21).

23. Device according to Claim 22, characterized by the fact that the multiplexer (19) contains electromechanical or electroöptical shutters that make it possible for the multiplexer to separately receive the light emitted by the light source (1), the polarized light R_t, R_s reflected by the sample (12) and the light T_r, T_s transmitted through the sample (12).

24. Utilization of the method according to Claim 1 for controlling the coating of substrates with thin layers.

25. Utilization of the method according to Claim 1 for controlling the reactive partial pressure during the coating of substrates.

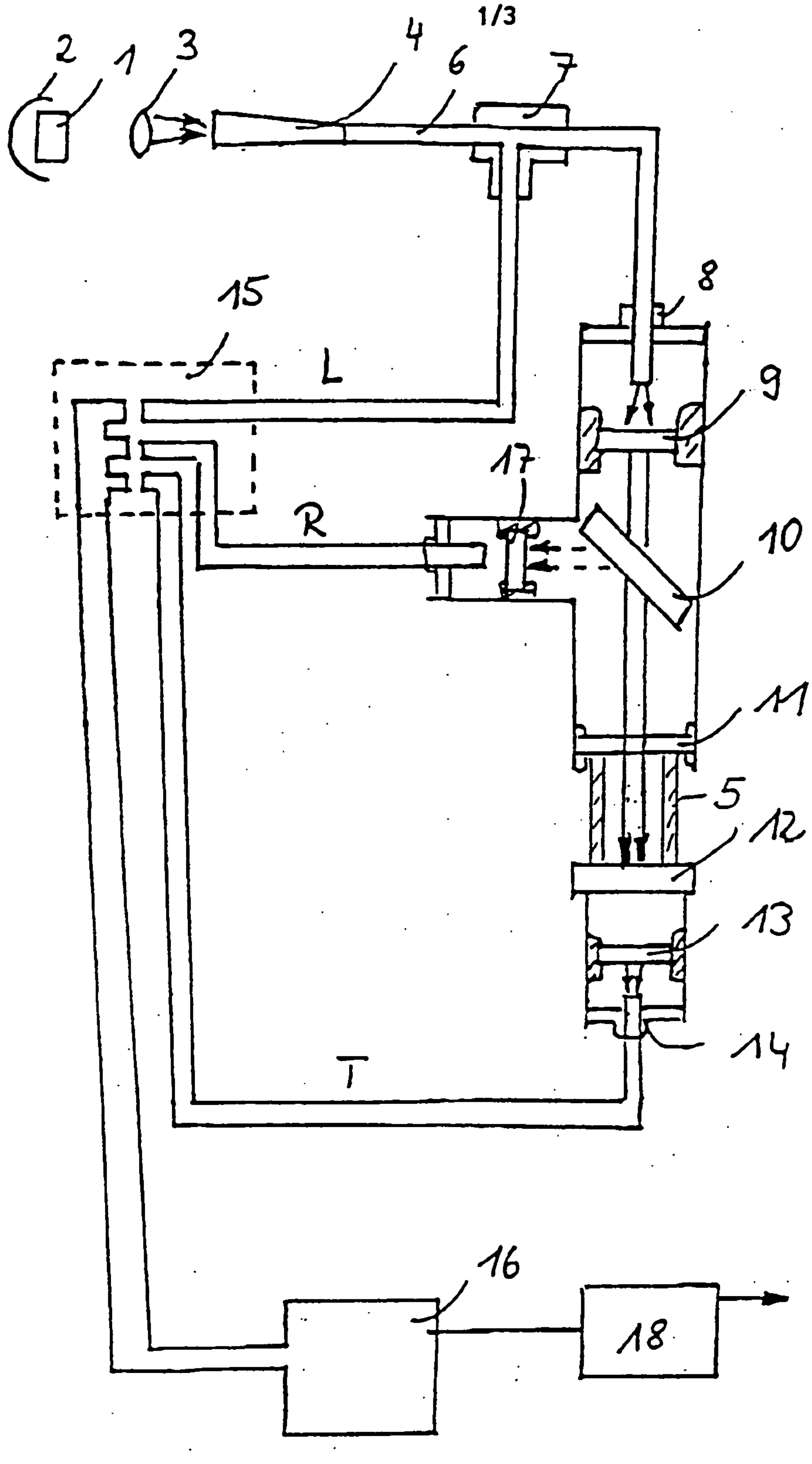

Figure 1

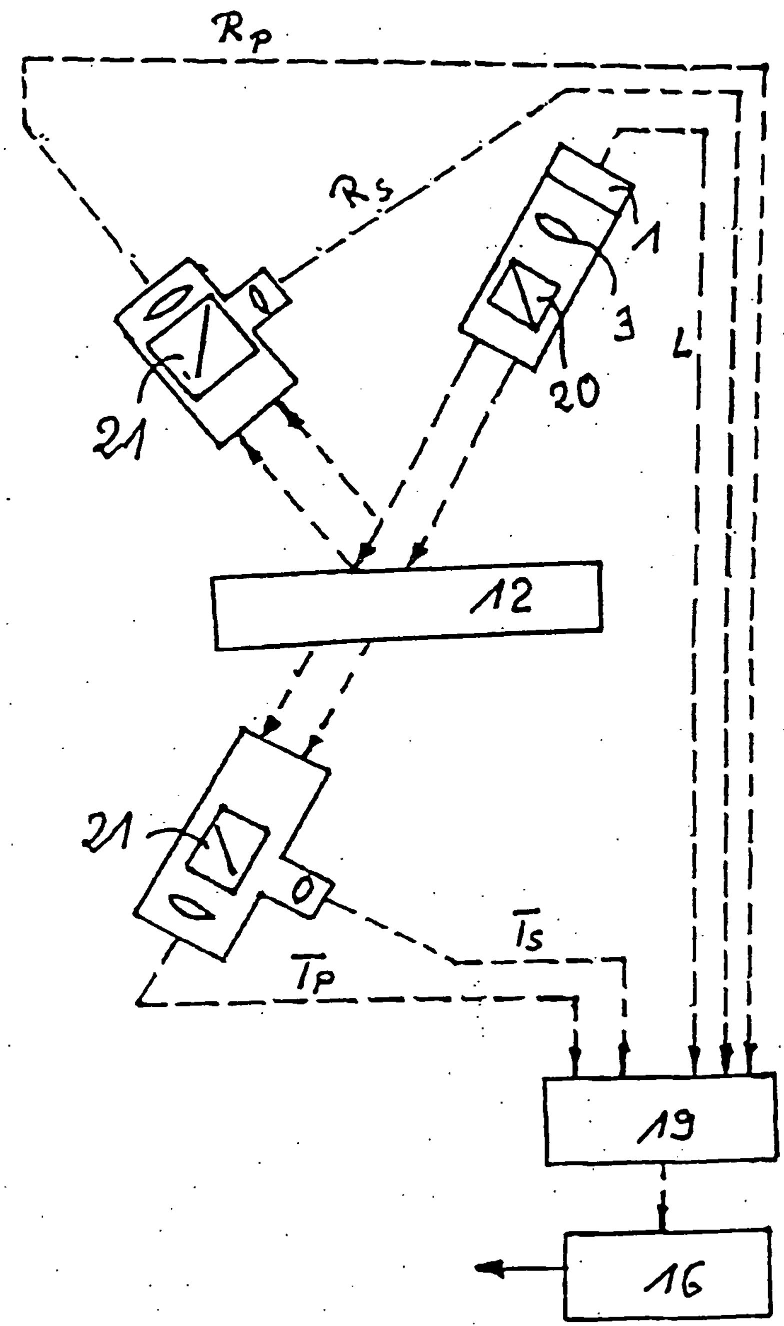

Figure 2

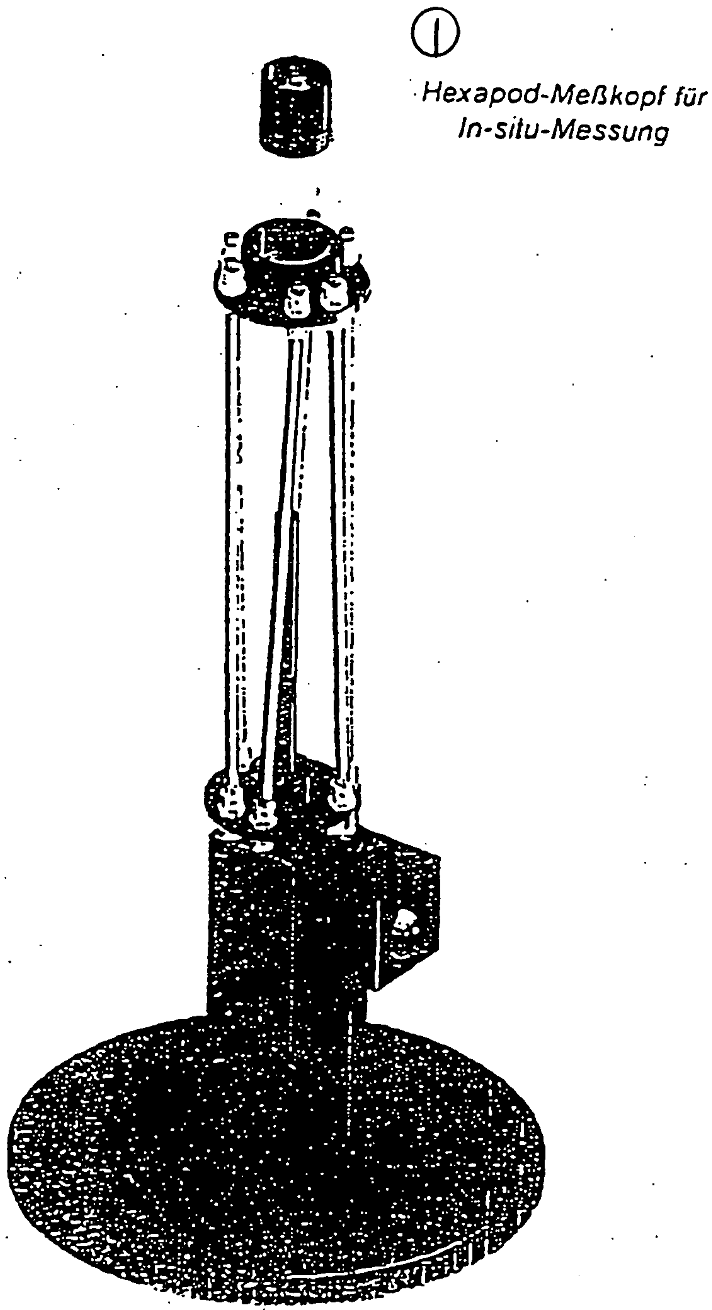

Figure 3

Key: 1 Hexapod measuring head for in-situ measurements

INTERNATIONAL SEARCH REPORT

Inte onal Application No
PCT/DE 96/01104

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 G01B11/06

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 G01B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X A	DE,A,32 34 534 (M.A. ORLOV ET AL.) 10 May 1984 1 siehe die gesamte Druckschrift ; see figures 1-3 ---	1-3,5-9, 15,17, 21,24 4,25
X	DE,A,32 48 091 (LEYBOLD HERAEUS GMBH) 28 June 1984 1 Siehe die gesamte Druckschrift ; see figures 1,2 --- -/--	1-9, 15-21, 24,25

[X] Further documents are listed in the continuation of box C.

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1

Date of the actual completion of the international search	Date of mailing of the international search report
18 September 1996	02.10.96

Name and mailing address of the ISA	Authorized officer
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[Key to previous page and the following page:]

1 See entire document
2 See introduction

Inte nal Application No
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US,A,4 776 695 (HUNG VAN PHAM, W.K. BORGIUM, C. MALLORY.) 11 October 1988 2 siehe die Einführung ; see column 3, line 51 - column 5, line 9 see column 6, line 38 - column 6, line 43; figures 1,3,5 ---	1-11,14, 17-19,24 1-11, 17-19,24
X Y	EP,A,0 257 229 (LEYBOLD-HERAEUS GMBH) 2 March 1988 1 Siehe die gesamte Druckschrift ; see figures 1-18 -----	1-12,15, 17-19, 21,24 1-11, 17-19,24

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/DE 96/01104

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